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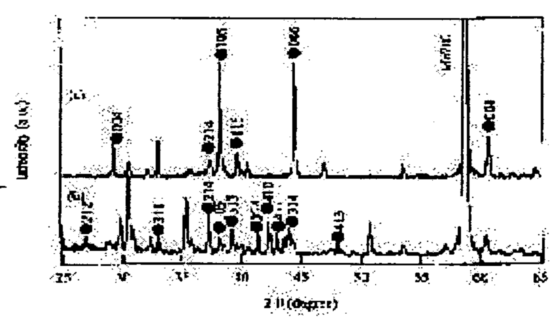
(54) R-Fe-B VERTICAL MAGNETIC ANISOTROPY THIN-FILM MAGNET AND MANUFACTURING METHOD THEREOF

(57)Abstract:

PROBLEM TO BE SOLVED: To provide an R-Fe-B vertical magnetic anisotropy thin-film magnet, which can be sharply improve both of its coercive force and its residual magnetic flux density by a film-forming method using a sputtering method, and to provide a manufacturing method of the magnet.

SOLUTION: A substrate is subjected to sputtering at normal temperatures, without heating the substrate on a composite board consisting of a heat-resistant metallic material of thermal conductivity higher than 50 W.m-1.K-1 at normal temperatures and a low-heat conduction material of a thermal conductivity lower than 1.1 W.m-1.K-1 at normal temperatures and after an R-Fe-B alloy thin film is formed, heat treatment is conducted on the alloy thin film or after a protective film is formed on the formed alloy thin film, heat treatment is conducted on the protective film.



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CLAIMS

[Claim(s)]
[Claim 1] The R-Fe-B system perpendicular magnetic-anisotropy thin film magnet which was formed in ordinary temperature sputtering and heat-treated after membrane formation on the compound substrate with which thermal conductivity [in / in the thermal conductivity in ordinary temperature / a -1-K-1 or more 50 W-m heat-resistant metallic material and ordinary temperature] consists of a -1-K-1 or less 1.1 W-m low-fever conduction ingredient and which has a perpendicular magnetic anisotropy to a film surface.
[Claim 2] The R-Fe-B system perpendicular magnetic-anisotropy thin film magnet which sequential membrane formation of the R-Fe-B system alloy film and the protective coat was carried out in ordinary temperature sputtering, and was heat-treated after membrane formation on the compound substrate with which thermal conductivity [in / in the thermal conductivity in ordinary temperature / a -1-K-1 or more 50 W-m heat-resistant metallic material and ordinary temperature] consists of a -1-K-1 or less 1.1 W-m low-fever conduction ingredient and which has a perpendicular magnetic anisotropy to a film surface.
[Claim 3] The manufacture approach of a R-Fe-B system perpendicular magnetic-anisotropy thin film magnet including the process to which the thermal conductivity in ordinary temperature forms a R-Fe-B system alloy in ordinary temperature sputtering on the compound substrate with which the thermal conductivity in a -1-K-1 or more 50 W-m heat-resistant metallic material and ordinary temperature consists of a -1-K-1 or less 1.1 W-m low-fever conduction ingredient, and the process which heat-treats on the R-Fe-B system alloy alloy film which formed membranes.
[Claim 4] The process to which the thermal conductivity in ordinary temperature forms a R-Fe-B system alloy in ordinary temperature sputtering on the compound substrate with which the thermal conductivity in a -1-K-1 or more 50 W-m heat-resistant metallic material and ordinary temperature consists of a -1-K-1 or less 1.1 W-m low-fever conduction ingredient, The manufacture approach of a R-Fe-B system perpendicular magnetic-anisotropy thin film magnet including the process which forms a protective coat on the R-Fe-B system alloy film which formed membranes, and the process which heat-treats on the R-Fe-B system alloy alloy film which has a protective coat.

[Translation done.]

* NOTICES *

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the thin film magnet used for a micro motor, a micro-actuator, a micro magnetometric sensor, etc., the R-Fe-B system perpendicular magnetic-anisotropy thin film magnet which has high coercive force and a high residual magnetic flux density, and has a perpendicular magnetic anisotropy to a film surface especially, and its manufacture approach.
[0002]
[Description of the Prior Art] In recent years, thin shape-ization of the magnet used for a motor, ata CHUETA, etc. is demanded with small and high-performance-izing of electronic equipment.
[0003] The R-Fe-B system permanent magnet which has high coercive force and a high residual magnetic flux density for current and these applications is used abundantly. Although a R-Fe-B system magnet is the ingredient optimal from the outstanding magnetic properties for thin-shape-izing, a sintered magnet and a bond magnet is [the thickness of about hundreds of micrometers] limitations.
[0004] Then, it has reported that research of thin-film-izing of a R-Fe-B system magnet was made, and got down, for example, as for Cadieu and others, coercive force obtained the thin film of 8-14kOe (637 - 1114 kA/m) by RF spatter in 1987 recently (6 Vac. Sci. Technol., A 1688 (1988)).
[0005] Moreover, Yamasaki and others has reported in 1990 that coercive force obtained the thin film of 3-7kOe (239 - 557 kA/m) by the DC magnetron sputtering method. Furthermore, Yamashita and others has got the thin film of coercive force 7kOe (557 kA/m) and residual magnetization 9.6kG (0.96T) by the DC magnetron sputtering method at the peak price in 1991 by the 65.5 to 77B10 to Nd13 - 17Fe17.5 presentation (the Magnetics Society of Japan 15,241-244 (1991)).
[0006]
[Problem(s) to be Solved by the Invention] By crystallizing depositing the film on the heated substrate, each above-mentioned approach tends to make the orientation growth of the c-axis of a crystal carry out in the perpendicular direction to a film surface, and tends to obtain perpendicular magnetic anisotropy films.
[0007] Although it is an approach simple for obtaining a thin film magnet, since the above-mentioned approach needs to heat a substrate, it has the problem that a substrate deteriorates or the structure of equipments, such as a problem of deforming, and temperature management, wiring of a substrate, becomes complicated, the problem that the soak nature of the substrate in heat treatment is still worse, etc.
[0008] Moreover, the coercive force of the obtained thin film is not yet practical. The highest in the inside of what is being cut by current is 14kOe(s) (1114 kA/m). When thermal resistance etc. is taken into consideration since the magnet operating point is low to the degree of pole in case a R-Fe-B system thin film magnet is put in practical use, 14kOe(s) (1114kA/(m)) are insufficient for coercive force, and it is desirable. [of a more high thing]
[0009] Artificers proposed the high coercive force R-Fe-B system thin film magnet which was formed in sputtering and was previously heat-treated after membrane formation on the substrate as a R-Fe-B system thin film magnet with high coercive force, without heating a substrate, and its manufacture approach (JP,11-288812,A).

[0010] However, since the thin film magnet in the above-mentioned proposal was isotropy, although it was excellent in ******, it had the problem that a residual magnetic flux density was small.
[0011] This invention aims at offer of the R-Fe-B system perpendicular magnetic-anisotropy thin film magnet which raised remarkably coercive force and a residual magnetic flux density and which has a perpendicular magnetic anisotropy to a film surface, and its manufacture approach in the membrane formation approach by ordinary temperature sputtering which does not heat a substrate for the purpose of offer of the R-Fe-B system thin film magnet with which are satisfied of both of high coercive force and a high residual magnetic flux density.
[0012]
[Means for Solving the Problem] In order that artificers may attain the above-mentioned purpose, as a result of inquiring wholeheartedly, the thermal conductivity in ordinary temperature on the compound substrate with which the thermal conductivity in a -1-K-1 or more 50 W-m heat-resistant metallic material and ordinary temperature consists of a -1-K-1 or less 1.1 W-m low-fever conduction ingredient After performing sputtering, without heating a substrate and making a R-Fe-B system alloy thin film form, By heat-treating on the optimal heat treatment conditions which can deposit a single domain particle from the amorphous thin film after necessary heat-of-crystallization processing, i.e., sputtering The knowledge of the thin film magnet which has a perpendicular magnetic anisotropy to a film surface, and has quantity ****** and a high residual magnetic flux density being obtained was carried out.
[0013] Furthermore, in order to prevent the oxidation at the time of heat treatment after R-Fe-B system thin film membrane formation, by carrying out membrane formation formation of the protective coat further on a R-Fe-B system alloy thin film, artificers did the knowledge of degradation prevention of magnetic properties being possible, and completed this invention.
[0014] The thermal conductivity in ordinary temperature this invention namely, on the compound substrate with which the thermal conductivity in a -1-K-1 or more 50 W-m heat-resistant metallic material and ordinary temperature consists of a -1-K-1 or less 1.1 W-m low-fever conduction ingredient It is the R-Pe-B system perpendicular magnetic-anisotropy thin film magnet which was formed in ordinary temperature sputtering, and was heat-treated after membrane formation, or protective coat **** Junji membrane formation was carried out with the R-Fe-B system alloy film in ordinary temperature sputtering, and was heat-treated after membrane formation and which has a perpendicular magnetic anisotropy to a film surface.
[0015] Moreover, the process to which, as for this invention, the thermal conductivity in ordinary temperature forms a R-Fe-B system alloy in ordinary temperature sputtering on the compound substrate with which the thermal conductivity in a -1-K-1 or more 50 W-m heat-resistant metallic material and ordinary temperature consists of a -1-K-1 or less 1.1 W-m low-fever conduction ingredient, It sets at the above-mentioned manufacture approach to the manufacture approach of a R-Fe-B system perpendicular magnetic-anisotropy thin film magnet including the process which heat-treats on the R-Fe-B system alloy alloy film which formed membranes, or a pan. It is the manufacture approach including the process which forms a protective coat on the R-Fe-B system alloy film which formed membranes of a R-Fe-B system perpendicular magnetic-anisotropy thin film magnet.
[0016]
[Embodiment of the Invention] In this invention, any well-known equipments, such as DC magnetron sputtering system usually used and RF sputtering system, can be used for sputtering. However, in this invention, since heating of a substrate is not needed, substrate heating apparatus etc. is not needed.
[0017] In this invention, as shown in the example mentioned later as target material for sputtering, the thing which dissolved and alloyed R, and Fe and B beforehand or the thing which has arranged each metal, for example, the thing which has arranged the chip of Nd and B on Fe plate, can be used.
[0018] What is necessary is just to determine the area which each metal in a target occupies so that it may be equivalent to the atomic ratio of the thin film magnet which it is going to obtain when arranging each metal which consists of R, Fe, and B and considering as a target. For

example, to the area of the whole target, if it is the becoming presentation Nd30Fe54B16, Nd will arrange each metal so that Fe may occupy and B may occupy 16% of area 54% 30%.
[0019] Moreover, as a presentation of a thin film magnet, all of a well-known R-Fe-B system alloy presentation are employable as the presentation list of a target. In order to aim at high coercive force, what contains R 20at(s)% - 30at%, and contains 10at(s)% - 16at% for B is desirable, and since residual magnetization will fall if ****** of B [less than / 20at% / and] does not improve [R] less than [10at%], but R exceeds and B exceeds 16at(s)% 30at(s)%, it is not desirable.
[0020] This invention is characterized by the thermal conductivity in ordinary temperature using the compound substrate with which the thermal conductivity in ordinary temperature serves as a -1-K-1 or more 50 W-m heat-resistant metallic material from a -1-K-1 or less 1.1 W-m low-fever conduction ingredient as a substrate which forms a R-Fe-B system alloy thin film. That is, it becomes possible by keeping the heat by ordinary temperature sputtering warm to a substrate to some extent with said compound substrate like before, rather than heating a substrate to obtain the R-Fe-B system perpendicular magnetic-anisotropy thin film magnet which the R-Fe-B system alloy film formed and heat-treated has a perpendicular magnetic anisotropy to a film surface, and has quantity ****** and a high residual magnetic flux density.
[0021] A compound substrate has the thing which carried out the laminating of the plate or sheet of each above-mentioned ingredient, or the desirable thing which carried out sputtering membrane formation of the heat-resistant metallic material at the low-fever conduction ingredient. Moreover, the above-mentioned compound substrate arranges a heat-resistant metallic material side, and is used for the side by which the R-Fe-B system alloy film is formed. Moreover, as for the thickness of a substrate, it is desirable that it is 1mm or less.
[0022] It is because less than 50 are [a value] insufficient as for amorphous-izing of the R-Fe-B system alloy film and it becomes impossible for it to acquire desired magnetic properties that the thermal conductivity in the ordinary temperature of a heat-resistant metallic material considered as -1-K-1 or more 50 W-m after heat-treating in the perpendicular magnetic-anisotropy list of the R-Fe-B system alloy film. Moreover, it limited to the heat-resistant metallic material for preventing deterioration and deformation of the substrate by heat, and controlling a reaction with the R-Fe-B system alloy film also in heat treatment after membrane formation. As a heat-resistant metallic material, metals or those alloys, such as Mo, Ta, W, and Fe, etc. are desirable.
[0023] Moreover, when the value exceeded 1.1, the thermal conductivity in the ordinary temperature of a low-fever conduction ingredient considered as -1-K-1 or less 1.1 W-m, because the heat insulation effect of the substrate mentioned above was not acquired but it became impossible to acquire desired magnetic properties after heat-treating in the perpendicular magnetic-anisotropy list of the R-Fe-B system alloy film. As a low-fever conduction ingredient, soda glass (0.75 W-m -1-K-1), lead glass (0.6 W-m -1-K-1), Pyrex (1.1 W-m -1-K-1), etc. are desirable.
[0024] In this invention, it is desirable to form protective coats, such as Ti film, on the R-Fe-B system alloy thin film which formed membranes on the substrate by sputtering, for the purpose of antioxidizing of a R-Fe-B system alloy thin film, as shown in an example.
[0025] That is, although it crystallizes by heat treatment since the R-Fe-B system alloy thin film after sputtering is in an amorphous condition, in the case of the heat-of-crystallization processing, R component in the oxygen in a heat treatment ambient atmosphere and a thin film etc. reacts, and there is a possibility that a thin film may oxidize. Therefore, by preparing a protective coat, oxidation of a thin film can be prevented and fall prevention of coercive force can be aimed at.
[0026] As a protective coat, Ti film shown in an example can be formed by the gaseous-phase forming-membranes methods, such as the sputtering method and vacuum deposition, and is a desirable example. The thickness of a protective coat is 100-1000A preferably several 10A - 1000A of numbers.
[0027] In this invention, the R-Fe-B system alloy film with which the protective coat was formed by the R-Fe-B system alloy film formed in ordinary temperature sputtering on the compound

substrate or the front face after membrane formation is in an amorphous condition, and the R2Fe14B ferromagnetism phase which has a perpendicular magnetic anisotropy from an amorphous phase to a film surface can be deposited by heat-treating for crystallization.
[0028] The heat treatment conditions of the well-known recrystallization according to the presentation of the R-Fe-B system alloy film etc. can adopt heat treatment. For example, as shown in the example mentioned later, it is desirable to carry out in the ambient atmosphere in a vacuum in temperature [of 550 degrees C - 700 degrees C] and time amount 30 minutes -, and 60 minutes because of oxidation control.
[0029] Although there is especially no problem also in inert gas, in order to lessen oxygen as much as possible, once making a heat treatment ambient atmosphere into a vacua, it is desirable [an ambient atmosphere] to take the technique of carrying out Ar permutation. Moreover, heat treatment temperature is not desirable in order that lifting coercive force may decline the grain growth which crystallization is not enough and exceeds 700 degrees C at less than 550 degrees C. Although influenced by the amount of the class and gestalt of the furnace to process, and a heat-treated object etc., heat treatment time amount can almost be crystallized, if it is usually 30 minutes - about 60 minutes.
[0030] This invention mentioned above enables it in the former to obtain the R-Fe-B system perpendicular magnetic-anisotropy thin film magnet which has the high coercive force and the high residual magnetic flux density which were not able to be obtained. specifically, the R-Fe-B system perpendicular magnetic-anisotropy thin film magnet which has the outstanding magnetic properties more than maximum energy product 127.3 kJ/m3 (16MGOe) is obtained more than residual magnetic flux density 0.8T (8.0kG) more than coercive force 1393 kA/m (17.5kOe).
[0031]
[Example] As a target, the presentation ratio prepared the disk with 5mm [in thickness], and a diameter of 100mm which consists of Nd20Fe 64B16 and which carried out casting, and fixed to the sputtering system. Moreover, as a substrate, Mo sheet with a thickness of 0.1mm and heat-resisting glass (Pyrex) with a thickness of 0.5mm were prepared, and the compound substrate fixed to the water-cooled substrate holder in a sputtering system in order of said heat-resisting glass and Mo sheet was used. Distance between a target and a compound substrate was set to 50mm, RF sputtering was performed on condition that a 266x10 to 6 Pa ultimate vacuum, 665x10 to 3 Pa Ar gas pressure, and high-frequency power 350W, without performing heating to a substrate, and the R-Fe-B system alloy film which consists of thickness of about 1 micrometer was obtained.
[0032] Next, after exchanging the target for Ti and forming Ti thin film of 300A thickness on this R-Fe-B system alloy film, 650 degrees C and heat treatment for 30 minutes were performed with the image heat treating furnace by the 399x10 to 6 Pa (266x10 to 4 Pa degree of vacuum under heat treatment) ultimate vacuum.
[0033] As an example substrate of a comparison, except using only Mo sheet, sputtering was performed on the same conditions as an example 1, and the R-Fe-B system alloy film which consists of thickness of about 1 micrometer was obtained.
[0034] The X diffraction result of the obtained thin film magnet is shown in drawing 1. The (a) line in drawing 1 is an example, and the (b) line is as a result of the example of a comparison. Although any film is crystallized completely and the Nd2Fe14 B phase deposits as a main phase so that clearly from drawing 1, as for the thin film magnet by this invention shown in (a) in drawing 1, Nd2Fe14 B phase is known by that the c-axis stacking tendency strong against a perpendicular direction is shown to the film surface, and a thin film has a perpendicular magnetic anisotropy to a film surface. It turns out to be it symmetrically that the thin film magnet of (b) in drawing 1 exists by the shape of polycrystal of non-orientation. In addition, subphases other than Nd2Fe14 B phase were also looked at by the diffraction peak.
[0035] Moreover, the hysteresis loop which impressed the magnetic field and measured the obtained thin film magnet up to a maximum of 20 kOe at the room temperature is shown in drawing 2. Drawing 2 (a) is an example and drawing 2 (b) is as a result of the example of a comparison. Moreover, as for the perpendicular measurement result of a direction, and the duplex slash in drawing, the notation of the reverse of T characters in drawing shows the

measurement result of an parallel direction to a film surface to a film surface. It turns out that the thin film magnet by this invention shown in drawing 2 (a) has a perpendicular magnetic anisotropy so that clearly from drawing 2. On the other hand, it turns out that the thin film magnet shown in drawing 2 (b) is a magnetic anisotropy within a field.
[0036] Moreover, the result of having measured the thin film magnet by this invention obtained in the example with the SQUID magnetization measuring instrument is shown in drawing 3. The magnetic field was impressed in the perpendicular direction to maximum 5T to the film surface. In addition, the hysteresis loop amends demagnetizing field and is shown. The perpendicular magnetic-anisotropy thin film magnets of this invention by the example were coercive force 1393 kA/m (17.5kOe), residual magnetic flux density 0.8T (8.0kG), and maximum energy product 127.3 kJ/m3 (16MGOe).
[0037] As shown in the above example, the perpendicular magnetic-anisotropy thin film magnet by this invention has the high coercive force and the high residual magnetic flux density which were not able to be obtained with an old thin film magnet, and is a utilizable enough property. Moreover, the further improvement can be expected by reducing volume ratios, such as subphases other than the main phase, by modification of sputtering conditions etc.
[0038]
[Effect of the Invention] According to this invention, without oxidizing a thin film magnet, a substrate's deteriorating and deforming, or the structure of equipment becoming complicated, the R-Fe-B system perpendicular magnetic-anisotropy thin film magnet which has high coercive force and a high residual magnetic flux density is obtained, further, by forming protective coats, such as Ti, on the R-Fe-B system alloy film, the oxidation at the time of heat treatment can be prevented, and degradation of magnetic properties can be prevented.
[0039] As mentioned above, a micro motor, a micro-actuator, the micro magnetometric sensor of the R-Fe-B system perpendicular magnetic-anisotropy thin film magnet by this invention, etc. are the optimal for the application as which the permanent magnet of a **** thin shape is required.

[Translation done.]

DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]
[Drawing 1] It is the chart Fig. showing the X diffraction result of a thin film magnet, and the case where the (a) line in drawing is an example and the (b) line is an example of a comparison is shown.
[Drawing 2] It is the graph which shows the magnetic properties of a thin film magnet, and the case where (a) is an example and (b) is an example of a comparison is shown.
[Drawing 3] It is the graph which shows the magnetic properties of the thin film magnet of an example.

[Translation done.]

* NOTICES *

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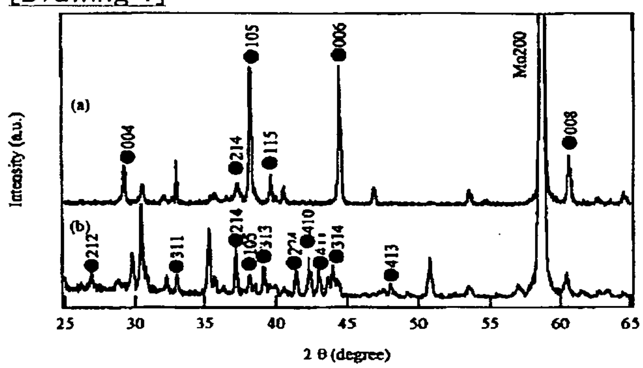
1.This document has been translated by computer. So the translation may not reflect the original precisely.
2.**** shows the word which can not be translated.
3.In the drawings, any words are not translated.

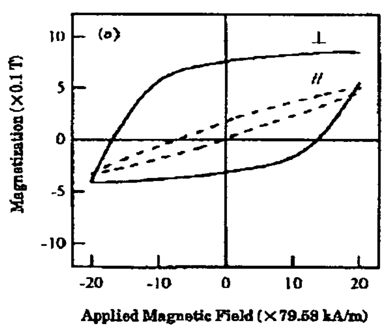
DRAWINGS

[Drawing 1]

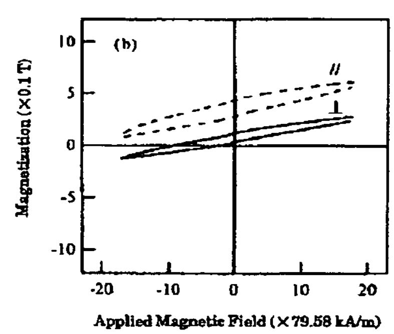


[Drawing 2]

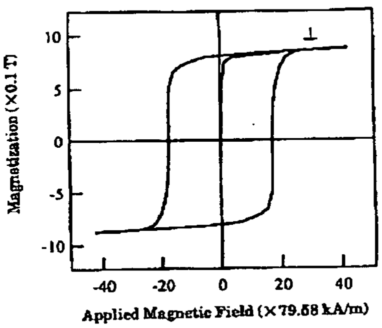
(a)



(b)



[Drawing 3]



[Translation done.]

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Ｃ２３Ｃ　14/14		Ｃ２３Ｃ　14/14	Ｆ
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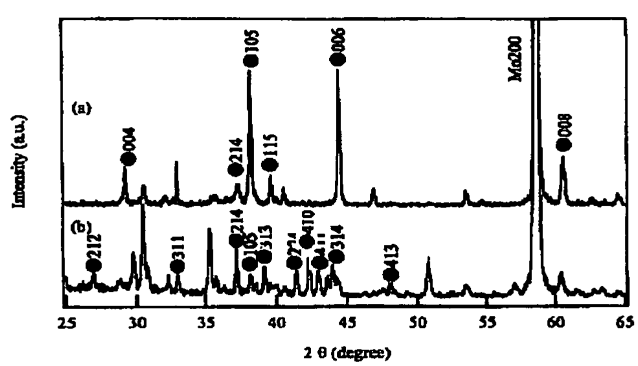
(54)【発明の名称】　Ｒ－Ｆｅ－Ｂ系垂直磁気異方性薄膜磁石及びその製造方法

(57)【要約】
【課題】　スパッタリングによる成膜方法において、保磁力と残留磁束密度の両方を著しく向上させることが可能なR－Fe－B系垂直磁気異方性薄膜磁石及びその製造方法の提供。
【解決手段】　常温における熱伝導率が50W・m^{-1}・K^{-1}以上の耐熱性金属材料と常温における熱伝導率が1.1W・m^{-1}・K^{-1}以下の低熱伝導材料とからなる複合基板上に、基板を加熱せずに常温スパッタリングを行い、R－Fe－B系合金薄膜を成膜した後熱処理を施すか、あるいは、成膜したR－Fe－B系合金膜上に保護膜を成膜した後熱処理を施す。



【特許請求の範囲】

【請求項1】　常温における熱伝導率が$50W\cdot m^{-1}\cdot K^{-1}$以上の耐熱性金属材料と常温における熱伝導率が$1.1W\cdot m^{-1}\cdot K^{-1}$以下の低熱伝導材料とからなる複合基板上に、常温スパッタリングにて成膜されかつ成膜後に熱処理された、膜面に対して垂直な磁気異方性を有するR－Fe－B系垂直磁気異方性薄膜磁石。

【請求項2】　常温における熱伝導率が$50W\cdot m^{-1}\cdot K^{-1}$以上の耐熱性金属材料と常温における熱伝導率が$1.1W\cdot m^{-1}\cdot K^{-1}$以下の低熱伝導材料とからなる複合基板上に、常温スパッタリングにてR－Fe－B系合金膜と保護膜が順次成膜されかつ成膜後に熱処理された、膜面に対して垂直な磁気異方性を有するR－Fe－B系垂直磁気異方性薄膜磁石。

【請求項3】　常温における熱伝導率が$50W\cdot m^{-1}\cdot K^{-1}$以上の耐熱性金属材料と常温における熱伝導率が$1.1W\cdot m^{-1}\cdot K^{-1}$以下の低熱伝導材料とからなる複合基板上にR－Fe－B系合金を常温スパッタリングにて成膜する工程、成膜したR－Fe－B系合金合金膜に熱処理を施す工程とを含むR－Fe－B系垂直磁気異方性薄膜磁石の製造方法。

【請求項4】　常温における熱伝導率が$50W\cdot m^{-1}\cdot K^{-1}$以上の耐熱性金属材料と常温における熱伝導率が$1.1W\cdot m^{-1}\cdot K^{-1}$以下の低熱伝導材料とからなる複合基板上にR－Fe－B系合金を常温スパッタリングにて成膜する工程、成膜したR－Fe－B系合金膜上に保護膜を成膜する工程、保護膜を有するR－Fe－B系合金合金膜に熱処理を施す工程とを含むR－Fe－B系垂直磁気異方性薄膜磁石の製造方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】この発明は、マイクロモータ、マイクロアクチュエータ、マイクロ磁気センサなどに用いられる薄膜磁石、特に、高保磁力及び高残留磁束密度を有し、膜面に対して垂直な磁気異方性を有するR－Fe－B系垂直磁気異方性薄膜磁石とその製造方法に関する。

【0002】

【従来の技術】近年、電子機器の小型・高性能化に伴い、モータやアタチュエータなどに使用される磁石の薄型化が要求されている。

【0003】現在、それら用途に高保磁力、高残留磁束密度を有するR－Fe－B系永久磁石が多用されている。R－Fe－B系磁石は、その優れた磁気特性から、薄型化には最適な材料ではあるが、焼結磁石、ボンド磁石共に数百μm程度の厚みが限界である。

【0004】そこで、最近、R－Fe－B系磁石の薄膜化の研究がなされおり、例えば、Cadieuらは、1987年に、RFスパッタ法により保磁力が8～14kOe(637～1114kA/m)の薄膜を得たことを報告している(Vac. Sci. Technol., A6,1688(1988))。

【0005】また、山崎らは、1990年に、DCマグネトロンスパッタ法により、保磁力が3～7kOe(239～557kA/m)の薄膜を得たことを報告している。さらに、1991年には、山下らが、DCマグネトロンスパッタリング法により、$Nd_{13\sim17}Fe_{65.5\sim77}B_{10\sim17.5}$組成で、最高値で、保磁力7kOe(557kA/m)、残留磁化9.6kG(0.96T)の薄膜を得ている(日本応用磁気学会誌15,241-244(1991))。

【0006】

【発明が解決しようとする課題】上記の方法は、いずれも加熱した基板上に膜を堆積しながら結晶化することにより、膜面に対して垂直な方向に結晶のc軸を配向成長させて垂直磁化膜を得ようとするものである。

【0007】上記の方法は、薄膜磁石を得るには簡便な方法ではあるが、基板を加熱することが必要であるため、基板が変質したり変形するなどの問題、また、基板の温度管理や配線など装置の構造が複雑になるという問題、さらに熱処理における基板の均熱性が悪いという問題などがある。

【0008】また、得られた薄膜の保磁力がいまだ実用的ではない。現在分かっているものの中での最高が14kOe(1114kA/m)である。R－Fe－B系薄膜磁石を実用化する際は、磁石動作点が極度に低いため、耐熱性などを考慮すると、保磁力は14kOe(1114kA/m)では足りず、より高いことが望ましい。

【0009】発明者らは、先に、高保磁力を有したR－Fe－B系薄膜磁石として、基板上に、基板を加熱することなくスパッタリングにて成膜されかつ成膜後に熱処理された高保磁力R－Fe－B系薄膜磁石とその製造方法を提案した(特開平11－288812号)。

【0010】しかし、上記提案における薄膜磁石は、等方性であることから、保能力には優れるものの、残留磁束密度が小さいという問題があった。

【0011】この発明は、高保磁力及び高残留磁束密度の両方を満足するR－Fe－B系薄膜磁石の提供を目的とし、基板を加熱することがない常温スパッタリングによる成膜方法において、保磁力及び残留磁束密度を著しく向上させた、膜面に対して垂直な磁気異方性を有するR－Fe－B系垂直磁気異方性薄膜磁石とその製造方法の提供を目的とする。

【0012】

【課題を解決するための手段】発明者らは、上記の目的を達成するため鋭意研究した結果、常温における熱伝導率が$50W\cdot m^{-1}\cdot K^{-1}$以上の耐熱性金属材料と常温における熱伝導率が$1.1W\cdot m^{-1}\cdot K^{-1}$以下の低熱伝導材料とからなる複合基板上に、基板を加熱せずにスパッタリングを行い、R－Fe－B系合金薄膜を形成させた後、所要の結晶化熱処理、すなわちスパッタリング後のアモルファス薄膜から、単磁区粒子を析出させることができる最適な熱処理条件で熱処理することにより、膜面に対して垂直な磁気異方性を有し、高保能力及び高残留磁束密度を有す

る薄膜磁石が得られることを知見した。

【0013】さらに、発明者らは、R-Fe-B系薄膜成膜後の熱処理時の酸化を防止するために、R-Fe-B系合金薄膜上にさらに保護膜を成膜形成することにより、磁気特性の劣化防止が可能であることを知見し、この発明を完成した。

【0014】すなわち、この発明は、常温における熱伝導率が50W・m^{-1}・K^{-1}以上の耐熱性金属材料と常温における熱伝導率が1.1W・m^{-1}・K^{-1}以下の低熱伝導材料とからなる複合基板上に、常温スパッタリングにて成膜されかつ成膜後に熱処理されるか、あるいは常温スパッタリングにてR-Fe-B系合金膜と保護膜力潮順次成膜されかつ成膜後に熱処理された、膜面に対して垂直な磁気異方性を有するR-Pe-B系垂直磁気異方性薄膜磁石である。

【0015】また、この発明は、常温における熱伝導率が50W・m^{-1}・K^{-1}以上の耐熱性金属材料と常温における熱伝導率が1.1W・m^{-1}・K^{-1}以下の低熱伝導材料とからなる複合基板上にR-Fe-B系合金を常温スパッタリングにて成膜する工程、成膜したR-Fe-B系合金合金膜に熱処理を施す工程とを含むR-Fe-B系垂直磁気異方性薄膜磁石の製造方法、あるいはさらに、上記製造方法において、成膜したR-Fe-B系合金膜上に保護膜を成膜する工程を含むR-Fe-B系垂直磁気異方性薄膜磁石の製造方法である。

【0016】

【発明の実施の形態】この発明において、スパッタリングには、通常用いられるDCマグネトロンスパッタリング装置、RFスパッタリング装置等、公知のいずれの装置も使用できる。但し、この発明においては、基板の加熱を必要としないため、基板加熱装置などは必要としない。

【0017】この発明において、スパッタリング用ターゲット材としては、後述する実施例に示す如く、予めRとFeとBを溶解し合金化したもの、あるいは個々の金属を配置したもの、例えば、Fe板上にNd及びBのチップを配置したものなどを用いることができる。

【0018】R、Fe、Bとからなる個々の金属を配置してターゲットとする場合、得ようとする薄膜磁石の原子比に相当するように、ターゲットにおける個々の金属が占める面積を決定すればよい。例えば、$Nd_{30}Fe_{54}B_{16}$なる組成であれば、ターゲット全体の面積に対して、Ndが30%、Feが54%、Bが16%の面積を占めるように各金属を配置する。

【0019】また、ターゲットの組成並びに薄膜磁石の組成としては、公知のR-Fe-B系合金組成のいずれをも採用できる。高保磁力を目的とするには、Rを20at%～30at%、Bを10at%～16at%を含有するものが好ましく、Rが20at%未満及びBが10at%未満では保協力が向上せず、Rが30at%、Bが16at%を超えると残留磁化が低下するため好ましくない。

【0020】この発明は、R-Fe-B系合金薄膜を成膜する基板として、常温における熱伝導率が50W・m^{-1}・K^{-1}以上の耐熱性金属材料と、常温における熱伝導率が1.1W・m^{-1}・K^{-1}以下の低熱伝導材料とからなる複合基板を用いることを特徴とする。すなわち、従来のように、基板を加熱するのではなく、前記複合基板で常温スパッタリングによる熱をある程度基板に保温することによって、成膜、熱処理されたR-Fe-B系合金膜が膜面に対して垂直な磁気異方性を有し、かつ高保能力、高残留磁束密度を有するR-Fe-B系垂直磁気異方性薄膜磁石を得ることが可能になる。

【0021】複合基板は、上記各材料の板材あるいはシートを積層したもの、あるいは低熱伝導材料に耐熱性金属材料をスパッタリング成膜したものなどが好ましい。また、上記複合基板は、R-Fe-B系合金膜が成膜される側に耐熱性金属材料面を配置して用いる。また、基板の厚みは1mm以下であることが望ましい。

【0022】耐熱性金属材料の常温における熱伝導率が50W・m^{-1}・K^{-1}以上としたのは、値が50未満では、R-Fe-B系合金膜のアモルファス化が不十分で、R-Fe-B系合金膜の垂直磁気異方性並びに熱処理後に所望の磁気特性を得ることができなくなるためである。また、耐熱性金属材料に限定したのは、熱による基板の変質・変形を防止し、かつ成膜後の熱処理においてもR-Fe-B系合金膜との反応を抑制するためである。耐熱性金属材料としては、Mo、Ta、W、Feなどの金属あるいはそれらの合金などが好ましい。

【0023】また、低熱伝導材料の常温における熱伝導率が1.1W・m^{-1}・K^{-1}以下としたのは、値が1.1を超えると、上述した基板の保温効果が得られず、R-Fe-B系合金膜の垂直磁気異方性並びに熱処理後に所望の磁気特性を得ることができなくなるためである。低熱伝導材料としては、ソーダガラス(0.75W・m^{-1}・K^{-1})、鉛ガラス(0.6W・m^{-1}・K^{-1})、パイレックス(1.1W・m^{-1}・K^{-1})などが好ましい。

【0024】この発明において、R-Fe-B系合金薄膜の酸化防止を目的に、スパッタリングにより基板上に成膜したR-Fe-B系合金薄膜上に、実施例に示すごとく、Ti膜等の保護膜を形成することが好ましい。

【0025】すなわち、スパッタリング後のR-Fe-B系合金薄膜はアモルファス状態であるため、熱処理によって結晶化するが、その結晶化熱処理の際に、熱処理雰囲気中の酸素と薄膜中のR成分などが反応し、薄膜が酸化する恐れがある。そのため、保護膜を設けることにより、薄膜の酸化を防止し、保磁力の低下防止を図ることができる。

【0026】保護膜として、例えば実施例に示すTi膜は、スパッタリング法や蒸着法などの気相成膜法により形成することができ、好ましい一例である。保護膜の厚みは、数10Å～数1000Å、好ましくは100～1000Åである。

【0027】この発明において、複合基板上に、常温スパッ

タリングにて成膜されたR-Fe-B系合金膜あるいは成膜後表面に保護膜が成膜されたR-Fe-B系合金膜はアモルファス状態にあり、結晶化のための熱処理を施すことにより、アモルファス相から膜面に対して垂直な磁気異方性を有する$R_2Fe_{14}B$強磁性相を析出させることができる。

【0028】熱処理は、R-Fe-B系合金膜の組成等に応じた公知の再結晶化の熱処理条件が採用可能である。例えば、後述する実施例に示すごとく、酸化抑制のため、真空中雰囲気で、温度550℃～700℃、時間30分～60分で行なうことが好ましい。

【0029】熱処理雰囲気は、不活性ガス中でも特に問題はないが、酸素を極力少なくするために、一旦真空状態にした後、Ar置換する手法をとることが望ましい。また、熱処理温度は、550℃未満では結晶化が十分でなく、また700℃を超えると粒成長を起こし保磁力が低下するため好ましくない。熱処理時間は、処理する炉の種類や形態、被熱処理物の量などによって左右されるが、通常30分～60分程度であればほとんど結晶化することができる。

【0030】上述したこの発明により、従来では得ることができなかった、高保磁力及び高残留磁束密度を有するR-Fe-B系垂直磁気異方性薄膜磁石を得ることが可能となる。具体的には、保磁力1393kA/m(17.5kOe)以上、残留磁束密度0.8T(8.0kG)以上、最大エネルギー積127.3kJ/m^3(16MGOe)以上もの優れた磁気特性を有するR-Fe-B系垂直磁気異方性薄膜磁石が得られる。

【0031】

【実施例】ターゲットとして、組成比が$Nd_{20}Fe_{64}B_{16}$からなる、鋳込成形した厚さ5mm、直径100mmの円板を準備し、スパッタリング装置に固定した。また、基板として、厚さ0.1mmのMoシートと厚さ0.5mmの耐熱ガラス(パイレックス)を準備し、スパッタリング装置内の水冷基板ホルダに前記耐熱ガラス、Moシートの順で固定した複合基板を用いた。ターゲットと複合基板間の距離を50mmとし、到達真空度266×10^{-6} Pa、Arガス圧665×10^{-3} Pa、高周波電力350Wの条件で、基板への加熱を行わずにRFスパッタリングを行い、厚み約1μmからなるR-Fe-B系合金膜を得た。

【0032】次に、ターゲットをTiに交換して、該R-Fe-B系合金膜上に、300Å厚みのTi薄膜を成膜した後、イメージ熱処理炉で、到達真空度399×10^{-6} Pa(熱処理中の真空度266×10^{-4} Pa)で650℃、30分間の熱処理を施した。

【0033】比較例

基板として、Moシートのみを用いる以外は実施例1と同じ条件でスパッタリングを行い、厚み約1μmからなるR-Fe-B系合金膜を得た。

【0034】得られた薄膜磁石のX線回折結果を図1に示す。図1内の(a)線が実施例、(b)線が比較例の結果である。図1から明らかなように、いずれの膜も完全に結晶化しており、$Nd_2Fe_{14}B$相が主相として析出しているが、図1内の(a)に示すこの発明による薄膜磁石は、$Nd_2Fe_{14}B$相は膜面に対して垂直な方向に強いc軸配向性を示しており、薄膜が膜面に対して垂直な磁気異方性を有することが分かる。それとは対称的に、図1内の(b)の薄膜磁石は、無配向の多結晶状で存在していることが分かる。なお、回折ピークには、$Nd_2Fe_{14}B$相以外の副次相も見られた。

【0035】また、得られた薄膜磁石を、室温で最大20kOeまで磁場を印加して測定したヒステリシスループを図2に示す。図2(a)が実施例、図2(b)が比較例の結果である。また、図中の逆T字の記号は膜面に対して垂直な方向の測定結果、図中の二重斜線は膜面に対して平行な方向の測定結果を示す。図2から明らかなように、図2(a)に示すこの発明による薄膜磁石は垂直磁気異方性を有することが分かる。一方、図2(b)に示す薄膜磁石は、面内磁気異方性であることが分かる。

【0036】また、実施例にて得られたこの発明による薄膜磁石を、SQUID磁化測定器により測定した結果を図3に示す。磁場は膜面に対して垂直な方向に最大5Tまで印加した。なお、ヒステリシスループは反磁場を補正して示してある。実施例によるこの発明の垂直磁気異方性薄膜磁石は、保磁力1393kA/m(17.5kOe)、残留磁束密度0.8T(8.0kG)、最大エネルギー積127.3kJ/m^3(16MGOe)であった。

【0037】以上の実施例に示すように、この発明による垂直磁気異方性薄膜磁石は、これまでの薄膜磁石では得ることができなかった高保磁力、高残留磁束密度を有しており、十分実用化できる特性である。また、スパッタリング条件などの変更によって、主相以外の副次相などの体積比を低減することにより、さらなる向上が見込める。

【0038】

【発明の効果】この発明によれば、薄膜磁石を酸化させたり、基板が変質・変形したり、装置の構造が複雑になることなく、高保磁力及び高残留磁束密度を有するR-Fe-B系垂直磁気異方性薄膜磁石が得られ、さらに、R-Fe-B系合金膜上に、Tiなどの保護膜を成膜することにより、熱処理時の酸化を防止し、磁気特性の劣化を防止することができる。

【0039】以上のように、この発明によるR-Fe-B系垂直磁気異方性薄膜磁石は、マイクロモータ、マイクロアクチュエータ、マイクロ磁気センサなど、極く薄型の永久磁石が要求される用途に最適である。

【図面の簡単な説明】

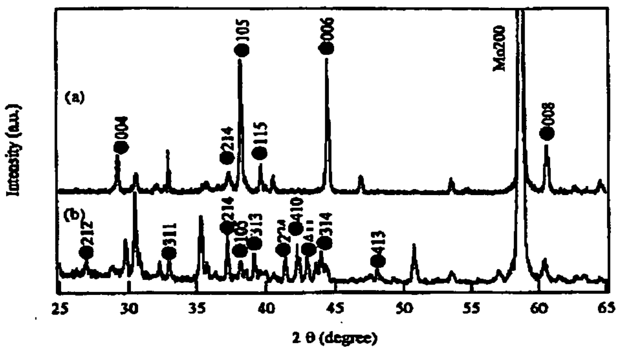
【図1】薄膜磁石のX線回折結果を示すチャート図であり、図中の(a)線が実施例、(b)線が比較例の場合を示す。

【図2】薄膜磁石の磁気特性を示すグラフであり、(a)が実施例、(b)が比較例の場合を示す。

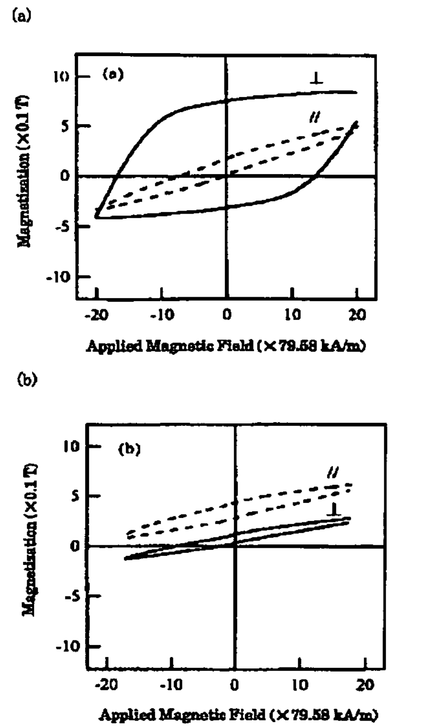
【図3】実施例の薄膜磁石の磁気特性を示すグラフであ

る。

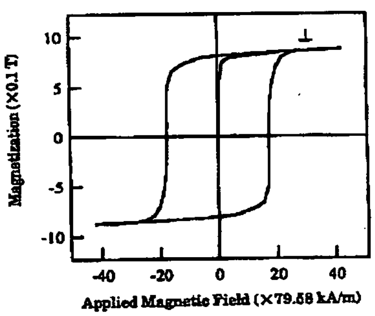
【図１】



【図２】



【図３】



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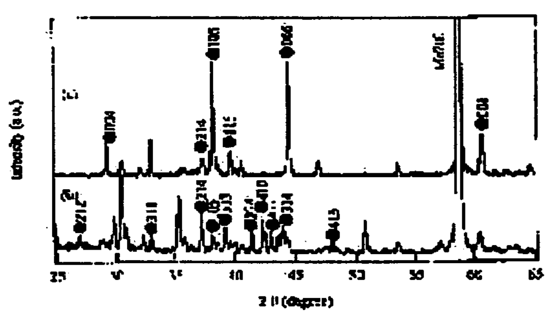
NAKANISHI AKIO

(54) R-Fe-B VERTICAL MAGNETIC ANISOTROPY THIN-FILM MAGNET AND MANUFACTURING METHOD THEREOF

(57)Abstract:
PROBLEM TO BE SOLVED: To provide an R-Fe-B vertical magnetic anisotropy thin-film magnet, which can be sharply improve both of its coercive force and its residual magnetic flux density by a film-forming method using a sputtering method, and to provide a manufacturing method of the magnet.
SOLUTION: A substrate is subjected to sputtering at normal temperatures, without heating the substrate on a composite board consisting of a heat-resistant metallic material of thermal conductivity higher than 50 W.m-1.K-1 at normal temperatures and a low-heat conduction material of a thermal conductivity lower than 1.1 W.m-1.K-1 at normal temperatures and after an R-Fe-B alloy thin film is formed, heat treatment is conducted on the alloy thin film or after a protective film is formed on the formed alloy thin film, heat treatment is conducted on the protective film.



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